Performance Monitoring Protocol (QA/QC) for the Thermo TSQ GC/MS (EI/CI)

1 Scope

This document addresses the performance monitoring (QA/QC) of the Thermo Triple Stage Quadrupole (TSQ) Gas Chromatograph/Mass Spectrometer (GC/MS) (EI/CI) System. This document applies to personnel using the associated instrument(s)/equipment in the following discipline/category of testing: Explosives (chemistry) examinations performed at the Huntsville facility.

2 Principle

The Thermo TSQ GC/MS consists of a Trace GC and a Triple Stage Quadrupole (TSQ) Mass Spectrometer (MS). These two instruments work in tandem and are referred to as the TSQ. The instrument is configured with either an electron impact (EI) ionization or a chemical impact (CI) ionization source using an interchangeable ion source cartridge.

When the instrument is in EI mode, it is implied that an EI volume is being used and that the reagent gas is off. Alternatively, when in CI mode, it is implied that a CI volume is being used and the reagent gas is on. The instrument can also be used with a Solids Probe. Solids Probe is a sample introduction technique utilizing the mass spectrometer for analysis and can be in either EI or CI mode. Definitions and guidelines for following this protocol are outlined in the "General Instrument Maintenance Protocol."

3 Equipment/Materials/Reagents

a. Instrumentation - Thermo TSQ8000 EVO, Thermo Trace 1310 GC and data system with XCalibur software (or equivalent)

b. Autosampler - Thermo AI/AS1310 ""automated sampler, accessories, and software (or equivalent)

c. GC Column - Agilent DB-5MS, 30 m, 0.25 mm i.d., 0.25 μm film (or equivalent)

d. Carrier Gas - Helium, 99.99% (high purity or equivalent)

e. CI Reagent Gas - Methane, 99.99% (high purity or equivalent)

f. Chloroform (GC grade)

g. Methanol (Optima grade)

h. Lidocaine HCl (Sigma or equivalent)

i. Tributoxyethyl Phosphate (TBEP) (Chem Service or equivalent)

j. Perfluorotributylamine (PFTBA, FC-43) (Thermo or equivalent)

k. Analytical balance

l. Volumetric flask

m. Autosampler vials - 2 mL GC vials, crimp or screw top, with or without 100-500 µL inserts (Thermo or equivalent)

n. Injection port liners - 3 mm split/splitless, tapered, with or without glass wool (Thermo or equivalent)

o. Injection port septa - low-bleed 17 mm (Thermo or equivalent)

p. Autosampler syringes - 10 µL syringe (Thermo or equivalent)

q. Wash vials – 4 mL screw top without insert (Thermo or equivalent)

4 Standards and Controls

4.1 Testmix (0.05 mg/mL each of Lidocaine and TBEP)

The Testmix is used to assess daily operating performance, mass assignment, and continued integrity of the system. To prepare, weigh 5.8 mg Lidocaine HCl and 5 mg TBEP into a 100-mL volumetric flask. Bring to the mark with chloroform and mix well. Store the solution in the refrigerator. It has a shelf-life of two years. This preparation may be appropriately scaled up. Record stock solution preparations in the Reagent Log.

4.2 PFTBA Tuning Solution

The PFTBA tuning solution is used for tuning the mass spectrometer and verifying mass calibration. It is supplied by the instrument manufacturer and does not expire. It is stored in a glass container attached to the TSQ. Refill as needed.

5 Sampling

Not applicable.

6 Procedures

6.1 Daily Checks

The following steps are to be performed daily. Enter the appropriate information in the QA/QC log for tracking purposes.

a. Check to ensure that the GC wash vials are filled with methanol, the waste vials are empty, and all are in the appropriate positions.

b. Record the remaining disk space on the hard drive. Use Windows to verify that the hard disk has at least 100 MB of free disk space. Do not use if less than 100 MB remain.

c. Record the line pressure of the building helium supply (carrier gas). The regulator should read 50 psi or above. If it cannot maintain this pressure, contact the appropriate instrument support personnel. If the helium is supplied by a gas cylinder, record the tank pressure. Change the tank if less than 100 psi remain.

d. If using CI mode, record the tank pressure of the methane tank (reagent gas). Change the tank if less than 100 psi remain.

e. Check the Ion Gauge to ensure that there are no significant leaks in the system. Do not use if the pressure is higher than 1×10^{-4} torr with the reagent gas off.

f. Prepare instrument for EI mode or CI mode; insert the correct ion volume. In the software, open the appropriate method based upon the ionization mode required. ‘’Check that the reagent gas is ON with a value of 2.0 mL/min for CI mode and OFF for EI mode.

g. Perform an analysis of the Testmix. Open the appropriate Testmix instrument method (such as ‘TestmixEI’ or ‘TestmixCI’), and verify the parameters as listed in the ‘Instrumental Conditions’ section of this protocol. Set up a sequence, load the autosampler with a vial containing the Testmix, and start the analysis. Evaluate the results using the ‘Decision Criteria’ section of this protocol. If the results are acceptable, print the following information for the Testmix:

    - For EI: RIC of m/z 86, RIC of m/z 299, and TIC. Label the peaks with scan number and/or retention time.

- For CI: RIC of m/z 235, RIC of m/z 399, and TIC. Label the peaks with scan number and/or retention time.

- Complete mass spectrum of both Lidocaine and TBEP.

h. If all requirements are within specification, prepare the documentation as outlined in the "General Instrument Maintenance Protocol." If any requirements fail, contact the appropriate instrument support personnel.

6.2 As Needed Checks

The following steps are to be performed as needed based on system performance. Indicate completion in the appropriate log.

a. Replace the septum in the GC injection port.

b. Replace the liner within the GC injection port.

c. Check the GC syringe in the autosampler. Replace if needed.

d. Appropriate instrument support personnel: Tune the mass spectrometer. Perform the following procedure for both EI and CI modes:

1. Ensure the current tune is saved.

2. Perform a standard tune.

3. Manual optimization of parameters may be performed to fine-tune the MS.

4. Acquire a mass spectrum of the PFTBA:

   - For EI: Collect approximately 20 scans

   - For Positive Ion CI: Collect approximately 20 scans

   - For Negative Ion CI: Collect approximately 20 scans

5. Evaluate the results using the 'Decision Criteria' section of this protocol. If the results are acceptable, print the mass spectrum from each.

e. If all requirements are within specification, prepare the documentation as outlined in the "General Instrument Maintenance Protocol." If any requirements fail, the appropriate instrument support personnel will determine the corrective action to be taken.

7 Instrumental Conditions

Refer to the "General Instrument Maintenance Protocol" for procedures on minor deviations.

7.1 Gas Chromatograph

Oven

Initial Temp:	60°C
Initial Time:	2.0 min
Ramp:	35°C/min
Final Temp:	250°C
Hold Time:	10.0 min

Inlet/Injector

Inj Vol:	1.0 µL
Mode:	Splitless
Inlet Temp:	220°C

Column

Type:	DB-5(MS)
Length:	30 m
Diameter:	0.25 mm
Film Thickness:	0.25 µm
Flow Mode:	Constant Flow, Vacuum Compensation
Pressure:	1 mL/min
Carrier Gas:	Helium

7.2 Mass Spectrometer

Solvent Delay:	5.0 min
Scan Mode:	Full Scan
Scan Range:	50-500 m/z (EI) 100-500 m/z (CI)

Temperatures

Transfer Line:	280°C
Source:	185°C

8 Decision Criteria

8.1 Testmix

Verify the results of the Testmix.

a. In order for the instrument to be considered in good operating condition, both Lidocaine and TBEP should generate well-resolved, Gaussian-shaped peaks with baseline separation.

b. A SNR of 3:1 will be the minimum response necessary to consider a response a peak.

c. There should be no extraneous peaks in the chromatogram/TIC greater than 5% of the height of the tallest Testmix peak.

d. The retention times of Lidocaine and TBEP should not deviate by ±3% compared to previous runs of the Testmix.

e. Check for the correct mass assignments for the mass spectra:
- EI - Lidocaine ions 86 and 234 and TBEP ions 57, 199, and 299.
- CI - Lidocaine ion 235 and TBEP ions 299 and 399.

8.2 Tune

Verify the results of the tune. Compare the results of the tune to previous tune results. The following are typical PFTBA values for the TSQ. If the observed PFTBA peaks are outside the values listed below, the appropriate instrument personnel will determine the corrective action to be taken.

a. PFTBA Tune: Mass assignments for m/z 69, 219, 414, 502, and 614

b. Relative abundance:
- EI: 69 or 219 base peak, 414 and 502 present
- CI+: 414 base peak, 219, 69, and 614 present
- CI-: 633 or 452 base peak, 414 present

9 Calculations

Not applicable.

10 Measurement Uncertainty

Not applicable.

11 Limitations

Only properly trained personnel will perform duties involved in the operation, maintenance, or troubleshooting of this instrument.

12 Safety

Take standard precautions for the handling of all chemicals, reagents, and standards. Refer to the *FBI Laboratory Safety Manual* for the proper handling and disposal of all chemicals. Personal protective equipment should be used when handling any chemical and when performing any type of analysis. Many instrument components are held at temperatures of 250°C and higher. Precautions should be taken to prevent the contact of skin with heated surfaces and areas.

13 References

Manufacturer's Instrument Manuals for the specific models and accessories used.

"General Instrument Maintenance Protocol" (IOG 001) *Instrument Operations Group SOP Manual.*

"Gas Chromatograph General Maintenance Protocol" (IOG 002) *Instrument Operations Group SOP Manual.*

"Mass Spectrometer General Maintenance Protocol" (IOG 004) *Instrument Operations Group SOP Manual.*

FBI Laboratory Safety Manual.

Rev. #	Issue Date	History
0	10/04/18	New document which specifies instrument protocol for the Huntsville facility.

Approval Redacted - Signatures on File

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